Subsequent relating cases 1795/27, 7319/42, 18519/43

C643 21/2

Department of Patents.
Patents Act 1903-1924
Commonwealth of Australia.

No. 25,900/25

APPLICATION DATED

12th November, 1925.

*Applicant (Actual Inventor)* ... ... JOHN AINSWORTH RICE.
*Application and Complete Specification* .. Lodged 12th November, 1925.
*Application and Complete Specification Accepted* 25th *Oct.*, 1926 Acceptance Advertised (Sec. 59): 3 Nov., 1926.

Classes 18.3 . 18.5.

*No drawing.*

Cellular Cement

COMPLETE SPECIFICATION.

## "Improvements in porous or cellular cements and methods of making the same."

I, JOHN AINSWORTH RICE, of 1165 Arch Street, Berkeley, in the State of California, United States of America, Chemical Engineer, a citizen of the United States of America, hereby declare this invention, and the manner in which it is to be performed, to be fully described and ascertained in and by the following statement:—

The invention relates to porous or cellular cement and to methods of making the same, and in the present specification the word "cement" is used broadly to include mortar, grout, concrete, plaster, or any such material which hardens or sets when it dries. The purpose of the invention is to provide a cementitious material which when set will contain a number of voids or spaces, whereby the material will be light but structurally strong and fireproof, and will effectively insulate sound and heat. This cement may be preferred in slabs or blocks, or may be laid down by pouring and allowed to set in situ.

The invention resides in cementitious material containing voids or cells, produced by a foam or lather stirred into the cement. Proposals for the production of porous cementitious material have hitherto been made in which foam or a foam producing substance is added to the cement mixing fluid. The foam producing substance has consisted of mucilage (tangin) or soluble soaps such as resin-soda or resin-potash soaps, and additions of gelatine and formaldehyde have been proposed to increase the durability of the foam.

According to the present invention a foam-assisting agent consisting of an unsaponified resinous material dissolved in an organic solvent such as alcohol or acetone is added to a colloidal solution of a foam-producing agent containing a proteid material as its essential constituent, and a gas is incorporated into the solution to produce a foam which is added to the cement. Various substances can be used as foam-producing agents, including gelatinous substances, and apparently any protein substances in colloidal solution will serve to make the foam. Egg albumen, blood albumen and casein are specially suitable: The foam-assisting agent preferably consists of rosin dissolved in alcohol or acetone, with sufficient formalin (40% formaldehyde) added to cause the resin to disperse when added to an aqueous liquid. The precipitate of resin remains in suspension and when the solution is beaten to a froth, the resin becomes a part of the walls of the bubbles, protecting and strengthening them and acting as a foaming agent before the bubbles are formed, in that it lowers the surface tension of the liquid and adds to its



C.16394.—11/11/26.—75.—Price, 1s. 6d. post free.

viscosity. Other resins, such as copal bakelite varnish, and also such materials as cellulose and rubber, may be used instead of rosin. For the foaming agent the following formulae may be used:—

| | (i) | (ii) |
|---|---|---|
| Rosin | 20 grams. | 100 grams. |
| Acetone | 50 cubic centimetres | — |
| Alcohol | — | 335 cubic centimeters. |
| Formalin | 34.5 cubic centimeters. | 131 cubic centimeters. |

The proportions of acetone and formalin or of alcohol or other solvent and formalin must be carefully balanced both as regards each other and as regards the amount of resin. The resin should be dissolved in the solvent and the formalin should be added in small portions, bit by bit, the mixing container being meanwhile shaken, until the solution becomes slightly cloudy. It may be tested by dropping a drop or two into clear water. If the solution emulsifies at once and spreads all through the water, forming a milky dispersed precipitate, the proportion of resin to solvent and to the formalin is correct. If the precipitate does not mix with the water, more formalin or more solvent should be added drop by drop. Maximum diffusibility is indicated by the precipitate diffusing through the water without clotting.

The foaming agent described above is to be mixed as follows to make a lather:—

| | |
|---|---|
| Foaming agent | 0.10% |
| Gelatine or glue | 0.15% |
| Water | 99.75% |

These proportions may vary widely, but it is desirable to use as much water as possible so that the other materials will not interfere with the setting of the cement.

Although the described foaming agent is preferred, glycerole may be used about as follows:—

| | |
|---|---|
| Water | 98.5% |
| Gelatine | 1.0% |
| Glycerole | 0.5% |
| or | |
| Water | 98.3% |
| Gelatine | 1.0% |
| Formalin | 0.2% |
| Glycerole | 0.5% |

Successful experiments have been made with Spanish Bark usually known as soap bark, a well known glucoside. The glucoside (1 part) is steeped into 99 parts of water for twenty minutes and the resultant liquor can be easily beaten into a stiff froth, mixing successfully with cement. Other glucosides and saponines may be used, viz. yucca of several varieties, the amole plant, a local soap root which in appearance resembles an onion and the roots of the senega plant. Solutions from these glucosides may be used alone or mixed with a gelatinous solution, for more efficient foaming. As a further foaming agent, phenol may be mentioned, which tends to stiffen the froth when added to a solution of gelatine, and which also seems to prevent breaking of the bubbles when mixed with strongly alkaline cements. Phenol is also a preservative for the gelatine solution, and phenol derivatives, among them lysol may be used. The effect of phenol or a phenol derivative upon the setting of Portland cement, is quite marked and very beneficial, counteracting the tendency of the glue to delay setting of the cement. The following formula may be used in this connection.

| | |
|---|---|
| Gelatine | 0.25% |
| Phenol | 0.12% |
| Formalin | 0.12% |
| Solution (1% of glucoside) | 0.25% |
| Water | 99.25% |

The vapor of chloroform markedly stabilizes the foam and renders it more insoluble in water or in the cement mixture. Chloroform may be sprayed into the beating chamber before the solution is beaten, so that the lather is made in an atmosphere of chloroform vapor. A foam made in this manner can be washed with water before being used in making the cement, whereby any excess of soluble gelatine is removed. Apparently the chloroform hardens the walls of the bubbles by toughening the films so that they do not easily break or dissolve in alkaline cements.

Different methods may be used for mixing gas into the solution to produce the foam or lather. For instance air may be introduced under pressure in the bottom of the tank containing the solution, and strained through a suitable filter.

The following are examples of other mixtures for producing the foam.

Example 1. 4 volumes of formalin are added to 1,000 volumes of a 2% glue solu-

:oside. The gluco-
l into 99 parts of
s and the resultant
n into a stiff froth,
h cement. Other
may be used, viz.
:, the amole plant.
in appearance re-
roots of the senega
ese glucosides may
with a gelatinous
nt foaming. As a
henol may be men-
fen the froth when
elatine. and which
caking of the bub-
strongly alkaline
a preservative for
phenol derivatives,
e used. The effect
erivative upon the
it, quite marked
ite ding the ten-
lay setting of the
formula may be

.. 0.25%
.. 0.12%
.. 0.12%
oside) 0.25%
.. 99.25%

m markedly stabi-
s it more insoluble
mixture. Chloro-
the beating cham-
is beaten, so that
an atmosphere of
oam made in this
with water before
e cement, whereby
latine is removed.
orm hardens the
ughening the films
k or dissolve

be used for mixing
produce the foam
air may be intro-
the bottom of the
:ion, and strained

ples of other mix-
oam.

s of formalin are
f a 2% glue solu-

tion. Separately a solution of rosin and formalin is made as follows:—

(A) 100 weight parts of resin.
335 weight parts of alcohol
18 weight parts of formalin (40% formaldehyde)

or

(B) 20 parts of rosin
16 parts of acetone
34 parts of formalin.

About 6 parts of the solution A or B should be added to 100 parts (by volume) of the glue solution. The rosin solution should be added while violently agitating the glue solution to insure immediate dispersion or dilution of the rosin solution. The mixture is then beaten to produce a foamy body. In place of rosin and the several substitutes therefor already mentioned, cellulose acetate, soluble phenol formaldehyde, or rubber may be advantageously used.

Example 2. 1 part of finely powdered common rosin is mixed with 16 parts of water and while agitated, enough ammonia water or other alkali is added to dissolve the rosin. This solution may be added to the glue solution as above described, using about 1 part of the rosin solution to 15 or 20 parts of the glue solution. In both the examples just described the strength of the glue solution may be varied for while a 2% solution is described, various concentrates, say from 0.1% to 10.0% can be used, and there are various substitutes for the glue, including gelatine, casein, albumen, solutions of various soluble adhesives such as dextrine, molasses, glucose, syrup, gum tragacanth and starch (boiled in water).

Example 3. A solution of a cellulose derivative such as cellulose acetate in acetone is added to a solution of casein in alkali, say 1 weight part of 6% solution of cellulose acetate in acetone to a solution of 4 weight parts of casein 1,000 weight part of water (containing sufficient alkali such as ammonia to dissolve the casein). This solution is converted into a foam directly, or after adding 1/250 weight part of formalin. Soap bark, common soap, rosin soap or the like may be added to make the mixture foam more strongly.

Example 4. 1 weight part of very thick viscose is added to 400 weight parts of a 2% solution of glue and water, or of a 1% solution of casein in alkaline water. This mixture may be converted into foam directly, but preferably with rosin soap, soap or soap bark. Formalin may be added if desired.

Example 5. To the solution given in example 4 or to a plain solution of viscose containing about 0.02% cellulose are added 40 parts of a rosin solution consisting of 60 parts of water and 1 part of powdered rosin. While stirring, sufficient ammonia water is added to dissolve the rosin. Then a sufficient amount of a weak solution (say 5%) of aluminium chloride, aluminium sulphate, a ferric salt, or an acid such as sulphuric or hydrochloric, or other suitable precipitant, is added to precipitate about from 4/5 to 9/10 of the rosin as resinate or a fine precipitate of rosin acids. The precipitate tends to produce a stable and tenacious foam and the precipitant improves the setting of Portland cement by preventing or greatly impeding chemical reaction between the cement and the soluble resinates.

Example 6. 4 parts of starch and 100 parts of water are stirred and boiled, and allowed to cool. Separately, 1 part of finely powdered rosin is added to 30 parts of water containing ammonia water to dissolve the rosin (if desired, partially neutralizing with alum or the like). The two solutions are mixed, and diluted with five times their volume of water, and beaten into a foam.

Example 7. Sulphuric acid is added to the resinate solution, to neutralize it, forming a colloidal suspension of resin acids. In the present example and example 6, sulphuric acid may be used in sufficient quantity to produce a slow reddening of litmus paper.

Example 8. A lather is made according to any of the foregoing examples, say with glue and formalin, and to about 5 cubic feet of the lather are added about 20 cubic centimeters of a 50% solution of calcium chloride ($CaCl_2$). This mixture is then incorporated with the cement grout.

In all the examples where a soap (say a resinate) or a solution of a resinous body (say shellac, rosin, etc. in alcohol or other solvent) or other foam-assisting body is used, the amount of that constituent should be sufficient to reduce greatly the surface tension of the liquid, whereby it will foam better.

Having now fully described and ascertained my said invention, and the manner

in which it is to be performed, I declare that what I claim is:—

1. A method of making a porous cement product characterized by the fact that a foam-assisting agent, consisting of an unsaponified resinous material dissolved in an organic solvent such as alcohol or acetone, is added to a colloidal solution of a foam-producing agent containing a proteid material as its essential constituent, and a gas is incorporated into such solution to produce a foam which is added to a cement grout.

2. A method according to Claim 1, characterized by the fact that a small amount of the rosin is used in relation to a much larger amount of the proteid material.

3. A method according to either Claim 1 and 2, and wherein rosin is the resinous material used, and a dispersing agent such as formalin is added to the resinous solution in amount substantially less than the amount of rosin, whereby the formalin modifies the action of the rosin.



4. A method according to Claims 1, 2 or 3, in which the foam is produced by agitation of the resinous solution and the solution of the foam-producing agent in the presence of chloroform vapor.

5. A method according to either Claim 1 or 2, wherein the resinous material consists of rosin, shellac, copal or soluble phenol formaldehyde with or without the addition of rubber, celluloid, cellulose acetate or viscose together with the dissolved proteid substance such as albumen, gelatine or glue.

6. A method according to any of the preceding claims, in which the colloidal proteid solution contains ammonia.

7. The improved method of making cementitious material substantially as herein described.

Dated this 21st day of September, A.D. 1926.

ANTHONY J. CALLINAN.
Patent Attorney for Applicant.

Witness—James H. Anderson.



Printed and Published for the DEPARTMENT OF PATENTS, COMMONWEALTH OF AUSTRALIA,
by H. J. GREEN, Government Printer for the State of Victoria.